Comparison Study of Sawdust and Palm Kernel Shell (PKS) for Pellet Fuel

by

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15277

Dissertation submitted in partial fulfilment of

the requirements for the

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CERTIFICATION OF APPROVAL

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Approved by,

_____

(Noorfidza binti Yub Harun)

UNIVERSITI TEKNOLOGI PETRONAS
BANDAR SERI ISKANDAR, PERAK
September 2015

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources of persons.

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NOORSYAKIRAH BINTI CHE JALIR

ABSTRACT

The widespread availability of biomass, which is renewable and has no impact on global warming, has motivated extensive research in the past decade regarding the industrial development of thermochemical conversion plants. Biomass has the potential to play a significant role in the energy conversion decreasing the fossil fuel dependency. This study will compare the product gas produced (CH_4, CO_2, O_2 and H_2) by two different types of biomasses which are woody (sawdust) and agricultural (palm kernel shell, PKS). Another parameter that the author study is the effect of size of the particle which are less than 500 µm and less than 1000µm on the production of gas by using Gas Chromatography (GC) after conducting pyrolysis process by hydro-cracking reactor. Another objective is to analyze the concentration of product gas of pellets in hydro-cracking reactor (10g). The biomasses were characterized by instrumental analysis such as elementary analysis (CHNS-O), thermogravimetric analysis (TGA) and bomb-calorimeter. From TGA, three different regions can be distinguished from a TG curve and each range indicate different decomposition of biomass such as removal of moisture content, volatile matter and complete combustion. The result shows 10g of pellets tested in hydro-cracking reactor perform similar in the chemical characteristics and product gas, as compared to 10mg of powder using TG. The performance of PKS to be used in energy generation system is similar with the performance of sawdust, based on the product gas concentration. However, high ash content might contain high alkali matter or inorganic compound which may cause problem in the system.

ACKNOWLEDGEMENT

First and foremost, I would like to express my highest gratitude to Allah S.W.T. whom with His willing, I was able to complete another chapter of my success journey. Million thanks I wish to my parents, Che Jalir bin Hassan and Aminah binti Mohd Noor, who are very supportive. Their encouragement and motivation are the keys that strengthen me in enduring this challenging period of final year study.

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CHAPTER 1

INTRODUCTION

1.1 Background Study

Current energy scenario has shown that the total energy demand increases for 84% in developing countries compared to 49% for developed countries from 2007 to 2035 (Energy Information Administration). Studies also indicate 81.1% of world energy production is mainly depended on fossil fuel. Due to limited fossil fuel reserves, the increasing pace of world energy production is not the same as energy consumption.

Most of our energy comes from burning fossil fuels like petroleum, coal, and natural gas. Fossil fuels are attractive because people have learned to use them so effectively and they are relatively inexpensive and available (Judkins, 2000). However, energy conversion devices have contributed to the pollution of the atmosphere by the combustion of fossil fuels. When fossil fuels are burned, they release carbon dioxide and other gases which pollute the air we breathe and contribute to climate change.

As an alternative for power generation, presently in Malaysia, continuous efforts and researches are focusing on biomass since abundance of agricultural and forest residues are turned out to be not only useful energy but also applicable products (Mekhilef, Saidur & Safari, 2011). Biomass, which is grown on a sustainable basis is considered to be carbon neutral by most authorities. This is because the carbon dioxide released during combustion is balance by trees and energy crops that capture CO_2 (Robinson, 2014). However, high volume of biomass is needed due to its low energy content compared to fossil fuel.

There are several types of biomass used as energy conversion feedstock such as agricultural and forest product residues, wood, municipal solid waste, and industrial waste. One of the most common is in pellet form which can be made from various biomass materials, for example, excess wood from the saw mills. Pellets form is more preferable compared to direct use for many reasons. Industrial and domestic heating application has shown increasing interest in the production of biomass fuel pellet. Production is expected to triple to an estimated 46 million metric tons by 2020 as the market for fuel pellets is growing rapidly due to increase in demand (Harun & Afzal, 2015; Tilburg, 2013).

Thus, different pre-treatment methods are available including thermal, mechanical and chemical (Arias, 2008). This research will focus on pyrolysis, a process which biomass is heated in the presence of oxygen to produce primarily gaseous fuels, called producer gas that have many applications (Rajvanshi, 1986). It contains carbon monoxide, hydrogen, methane, benzene, toluene, CxHy aliphatic hydrocarbons and tars (besides CO2 and H2O). ~50% of the energy in the gas is made up of syngas components such as CO and H2. Methane and higher (aromatic) hydrocarbons stored the remainder of the energy (Boerrigter & Rauch, 2005).

A large array of methods and analytical systems is needed in the characterization of the conversion processes of lignocellulosic biomass to biofuels in order to describe the solid materials by extracting the necessary meaningful parameters. For instance, bomb-calorimeter is used to determine the sample's calorific value. Another analytical system that will be used in this study includes weight degradation of sample with respect to temperature by using Thermogravimetric Analysis (TGA), CHNS-O instrument to determine the elemental composition of the biomasses and last but not least to calculate the moisture content.

The focus of study is to compare the product gas concentration produced from both woody and agricultural biomasses. The prepared biomass will be pelletized before undergoes pyrolysis process and the gas produced at temperature 350°C,450°C and 540°C will be collected and analyzed by Gas Chromatography (GC). The parameter of interest is the product gas produced as the effect from different particle size during pelletization and different types of biomasses, woody and agricultural. This will indicate the potential of agricultural waste as a useful of fuel.

1.2 Problem Statement

In recent years, more attention has been paid to renewable energy, especially biomass energy. This is due to the fact that the increasing application of fossil fuels cause high CO_2, NO_x and SO_x emissions in the atmosphere and man fears their accompanying shortage occurring in the near future. Compared with fossil fuel, biomass is a renewable resource and has a much shorter period of life cycle (Chen et al., 2003).

According to (Tock, Lai, Lee & Tan, 2010) due to the implementation of energy policies and greater understanding on the importance of green energy, renewable energies such as hydropower and biomass in Malaysia are gaining popularity. With the availability of feedstock, since Malaysia has been well endowed with natural resources in areas such as agriculture and forestry that need to be utilized, biomass energy is practical to be conducted.

In developing countries that do not have strong implementation of regulatory instruments, biomass is left to rot or as a common practice, biomass is openly burned in the field. Direct combustion of agricultural residue results in air pollution and thus posing risk not only to ecological health but also human. Although utilization of biomass conversion technologies has shown an emerging trend, for instance, combustion of sugarcane bagasse and rice husk to gasification of other agricultural residues, biomass still largely under-utilized. The challenge of biomass is to convert biomass as a resource for energy and other productive uses (UNEP,2009). This is because although it is a renewable resource, it will cause problems when not used.

1.3 Objectives and Scope of Study

The main objectives of this study is as follows:

i. To conduct proximate and ultimate analysis of woody and agricultural biomass

ii. To compare degradation profile of two different sources of biomass

iii. To analyze the concentration of product gas (H_2, CO_2, CH_4, O_2) of pellets with two different particle sizes (<0.5mm and <1.0mm) in hydro-cracking reactor for 10g of pellets.

For the scope of study, it will include the pyrolysis of two types of biomass (woody and agricultural) using hydro-cracking reactor in an inert environment. The product gas will be collected at different temperatures by using gas bag. Then, the gas will be analyzed by GC. The product gas of both biomasses will be compared and analyzed for their concentration, specifically for H_2, CO_2, CH_4, and O_2.

The mass degradation with respect to temperature will be analyzed for each sample by using TGA. The ultimate analysis of the biomasses will also be conducted using CHNSO elemental analyzer. Sample's calorific value is determined by bomb-calorimeter instrument. Each individual biomass of different sizes (<1mm and <0.5mm) will be pelletized using pellet mills of about 200g for pyrolysis of 10g pellets in hydro-cracking reactor.

CHAPTER 2

LITERATURE REVIEW

2.1 Biomass in Malaysia

Malaysia is blessed with conventional energy resources such as oil and gas as well as renewables like solar, hydro, and biomass (Zafar, 2015). Located strategically in the middle of South East Asia, Malaysia is also gifted with fertile agriculture land (Fikri, 2013). This land provides great biomass and wood waste readily available for fast exploitation (Zafar, 2015) as shown in Figure 2.1. According to Malaysia Biomass Industries Confederation, the government has developed several strategies to develop and utilized the energy from biomass for instance, introduction of the National Green Technology Policy in 2009, the 10th Malaysia Plan (10MP) and the Economic Transformation Program (ETP) in 2010, as well as the Renewable Energy Act 2011.

Wood Chips

Forest Residues

Palm Kernel Shells

Rice Husk

FIGURE 2.1 Example of biomass feedstock

Each feedstock of biomass has its own chemical and physical properties and the characteristics vary significantly within and different biomass raw material. Malaysia needs to utilize the advantage of abundance forestry and agricultural biomass especially wood pellet. The demands of wood pellet are increasing because they are more favorable compared to other types of biomass (Biomass Energy Resource Centre, 2007). Wood pellets is one of biomass fuels that has gained national attention. Man has been burning wood as a form of energy for thousands of years and a lot of improvement has been explored. Comparison of woody and agricultural biomass is conducted since agricultural biomass also can contribute to pellet production in the market. For example, according to (Cerosaletti, 2011), grass energy can be a high energy fuel as shown in the Figure 2.2 below:

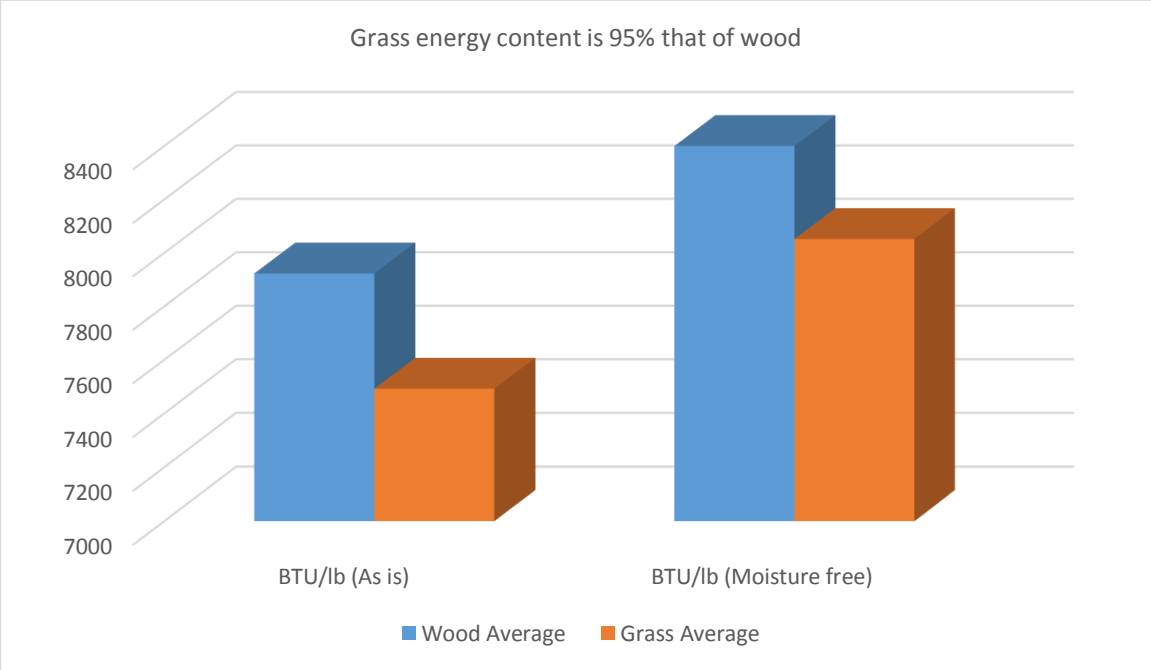

FIGURE 2.2 Comparison of Woody and Grass Energy Value

The selected biomass feedstock for wood material is saw dust, while for agricultural feedstock, the author chooses palm kernel shell (PKS). By referring to study conducted by (Abdullah & Yusup, 2010) regarding screening of Malaysian biomass based on their properties such as calorific value, moisture content, ash content, fixed carbon and volatile matter. Both biomasses show high calorific value,

fixed carbon and volatile matters, ash and moisture content. Palm kernel shell is selected amongst the palm oil wastes in Malaysia due to its properties such as high proportion of volatile matter and fixed carbon, low ash and moisture content (Abdullah et al., 2010). These make it suitable to be pelletized and undergoes pyrolysis process.

2.2 Advantages of Pelletized Biomass

Compared to the other biomass resources, wood pellets have their own specialty and advantages. According to (BioEnergy, 2013) pellets can be made from various biomass materials and one of the most common source is excess wood from the saw mills, or recycled pellets. Although there exists many other feedstock of biomass, wood pellets are more favorable as they are convenient and easy to use. Their energy content is high and highly efficient compared to other biomass (Missagia, 2011). They burn cleanly and are more convenient than logs or wood chip. Table 2.1 below shows the comparison of wood pellets and wood chips (Rolls, 2012):

TABLE 2.1 Comparison of Wood Pellets and Wood Chips

Wood pellets	**Wood Chips**
Moisture content, typically less than 10%	Moisture content typically ~55% from freshly harvested wood; `30% from seasoned wood (1 year)
Volume energy density is high: 3-3.5 MWh/m3	Volume energy density is relatively low (< 1 MWh.m3 @ 30% MC
Low dust, clean, dry. Flow easily so suitable for pneumatic delivery (up to 30m) and easy fuel handling.	Don't flow so readily. Delivery by tipper or chipping into store;
Usually low ash <1%	Can be low ash, but a high proportion of bark, or soil contamination can increase this significantly.
Summary	
Pleasant, convenient fuel and clean; more expensive but sophisticated boiler	Potentially suitable for self –production, bulky but cheap fuel
Relatively compact storage requirement	Boiler tend to be better suited to larger or agricultural type applications.

Furthermore, its moisture content is relatively reasonable for heating making it popular all over the world compared to some raw materials. According to (Woods Pellets Quality Standards [WPQS], 2015) the lower the moisture content of pellets, the higher utilization rate is since its primary purpose is for heating. In accordance with European Pellet Quality, the wood pellets will not be burned completely no matter for household or industrial if the moisture content is less than or equal to 10 w-% (WPQS,2015).

In combustion system, water content in the fuel must be driven off before the first stage of combustion can occur in order to reduce requiring of energy and thus can increase overall system efficiency and consequently increasing combustion temperature below the optimum. The emission of tars and creosote due reduction in combustion temperature below the optimum will occur as the result of incomplete combustion of the fuel (Bioenergy,2012). Table 2.2 below presented the various biomass moisture content ranges for different derivation raw materials.

TABLE 2.2 Typical Moisture Content of Various Biomass Sources

Biomass sources	**Moisture Content (calculated on wet basis)**
Wood chips	10-60%
Wood pellets	8-12%
Straw	20-30%
Sawdust	15-60%
Cotton stalks	10-20%
Switchgrass	30-70%
Chicken droppings	75-80%
Cheese whey	93-97%
Maize silage	65-75%

In addition, according to (Bootle, 2011), the advantages of pelletized biomass compared to other form of biomass are as follows:

i. Pellets are denser than chopped grass

ii. Lower transport costs

iii. Less frequent fuel deliveries

iv. Burns better

v. Easier to manage boiler

2.3 Characteristics of Pellets

Wood pellets is made by wood shreds under high pressure until the lignin softens and binds the material together or in simple word by compressing dry sawdust. Compared to wood chips, typically pellets gives greater volume energy up to three to four times that of wood chips resulting from combination of low moisture content (typically below 10%) and highly compressed material. They are particularly attractive in domestic application because they are clean, consistent and flow easily (Hogan, 2012).

For the pellets size, they should be produced uniformly, generally they are in cylinder-shaped. In this case, the diameter and length must be regular, which the diameter must be between 6-8 mm and the length ranges from 3.15-40 mm to comply with ENPlus standards (WPQS, 2015). This is supported by (European Biomass Industry Association [EBIA], 2012) that stated the pellets dimension should be 6mm in diameter and 25 mm length. The wrong size wood pellets may alter combustion conditions which will result in an increase in emissions and a loss of efficiency.

Wood pellets release much less ash compared with fossil fuels and ash content is one of the indicators of quality standards for wood pellets. According to Pellet Fuels Institute (PFI, 2012), the new standard about ash content requirements of wood pellets is as shown in Table 2.3.

TABLE 2.3 Standards and Ash Content Requirement

Standards	**Ash content requirement**
Super-Premium	<0.5% Ash
Premium	<1% Ash
Standard	<2% Ash
Utility	<6% Ash

When the pellet is being gasified, the resulting gas mixture is called producer gas which is actually a fuel. Since the gasified compounds were obtained from biomass, the power derived from the resultant gas is considered to be a source of renewable energy. The gas produced from the pyrolysis is the main focus of this study.

2.4 Biomass Conversion Technology

There are various conversion technologies available that can convert a wide variety of biomass resources as a renewable energy sources such as power, heat, and fuels. Conversion technologies may also convert it to another form such as combustible biogas or liquid biofuel and not only release the energy in the form of heat or electricity directly. Some classes of biomass may only applicable for one appropriate technology while for others there may be a number of usage options (Biomass Energy Centre,2011).

Four types of conversion technologies currently available such as thermal conversion, thermochemical conversion, biochemical conversion and chemical conversion which are appropriate for specific biomass types and resulting in specific energy products (Williams, 2011). Biomass conversion is mainly divided into two subgroups, thermal related process and biological related process (Ho, 2006) as shown in Figure 2.3 below.

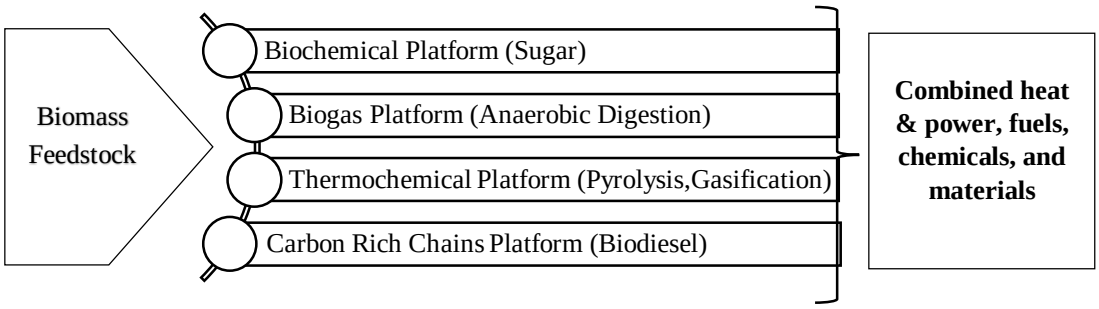

FIGURE 2.3 Biomass Conversion Technology

Processes such as direct combustion, pyrolysis and gasification is the example of thermal conversion which use heat, with or without the presence of oxygen in order to convert biomass materials or feedstocks into other forms of energy. The comparison of these processes as shown in the Table 2.4 below (Siirala, 2013).

TABLE 2.4 Comparison of Thermal Conversion

	Combustion	**Gasification**	**Pyrolysis**
Processes	-Occurs with sufficient oxygen to completely oxidize the fuel. -Temperature range: 800-1200°C.	-Occurs in insufficient oxygen or steam to avoid complete oxidation. -Temperature range: 800-1200°C.	-Occurs in the absence of oxygen. -Temperature range: 350-600°C.

Use	-Carried out in a boiler to generate steam, used for electricity production by steam turbine.	-Gas produced can be generated into electricity. Purified synthesis gas can be converted into chemicals as a feedstock.	-Bio-oils received by pyrolysis can be used as a source of fuel in combustion boiler, refined into transportation fuels or used as a chemical feedstock
Final Products	-Heat	-Heat & combustible gas -Char	-Bio-oil, gases, & heat -Char
Level of Technology	-Long known & widely used.	-Mature & been used for a long time. Already commercialized.	-Newer & not that widely used compared to combustion and gasification.

The products produced can be same but in a different ratio since combustion, pyrolysis and gasification have many similarities. The desired final products and end uses are to be taken into account when choosing the most suitable mechanism for energy production.

2.4.1 Pyrolysis

Pyrolysis is the precursor to gasification, consist of thermal decomposition in the absence of oxygen. It is a process of subjecting the biomass to high temperature (greater than 430°C) under pressurized environment and biomass will undergoes partial combustion (Williams, 2011). Pyrolysis takes place as part of gasification and combustion process. The product produced from this process such as gas, liquid and a

sold char will depend upon the parameters of the process. Figure 2.4 below shows simple representation of pyrolysis process (Brownsort, 2009).

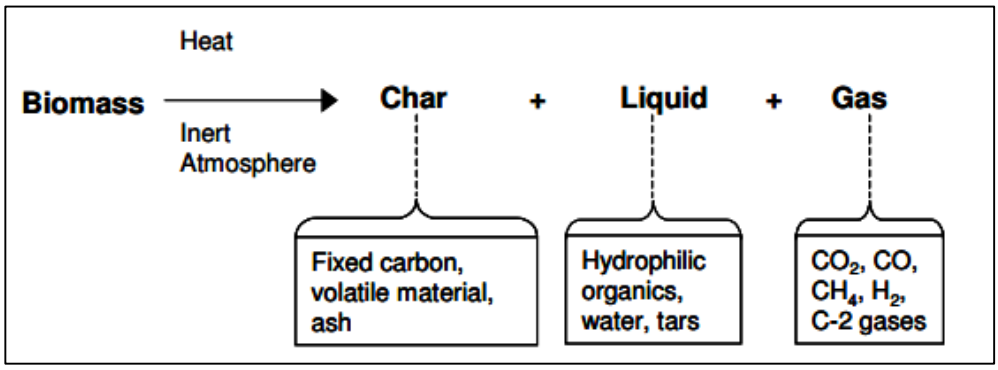

FIGURE 2.4 Pyrolysis Process

Pyrolysis can be classified into two main classes which are fast pyrolysis and slow pyrolysis. Fast pyrolysis is characterized by short vapor residence time and high heating rates. Small particle sizes are needed as a feedstock and a design that removes the vapors quickly from the presence of the hot solids (Williams, 2011). With temperatures between 300 and 550 degrees Celsius, pyrolysis systems take place in less than two seconds. Char must be removed frequently since it accumulates quickly in fast pyrolysis (BioEnergy,2011).

Examples on slow pyrolysis can be divided are traditional charcoal making and more modern processes. Typically, the temperature for slow pyrolysis is at 400°C and it is lower than fast pyrolysis. Slower heating rates, relatively long solid and vapor residence times are factors characterized the slow pyrolysis. The target product is often the char, but this will always be accompanied by liquid and gas products although these are not always recovered (Brownsort, 2009).

2.5 Product Gas

The gasification temperature plays a vital role in the utilization of gases from biomass gasification as different temperature will produce different gas. For instance,

biosyngas is generated at temperature >1200°C while a product gas is at <1000°C, of which the latter can be converted into biosyngas by catalytic or thermal cracking (Rauch, 2005). In the utilization for power generation and synthetic natural gas synthesis, product gas is preferred. Apart from temperature, the other parameter that affect the product gas is type of biomass used and size of particles.

The direct use for the generation of power (and heat) is the major application of product gas. This can be either in stand-alone combined heat and power (CHP) plants or by co-firing of the product gas in large-scale power plants. The production of synthetic natural gas (SNG) is the second major application of product gas. As a way of-co-combustion, the gas which is in H_2, CH_4, etc. with low ash and sulphur content is an ideal co-combustion fuel and effective to decrease nitrogen oxides emissions (Dahlquist, 2013). It also helps to avoid most of the problem associated direct co-combustion such as corrosion, boiler fouling and ash characteristics altering.

CHAPTER 3

METHODOLOGY

3.1 Project Flow Chart

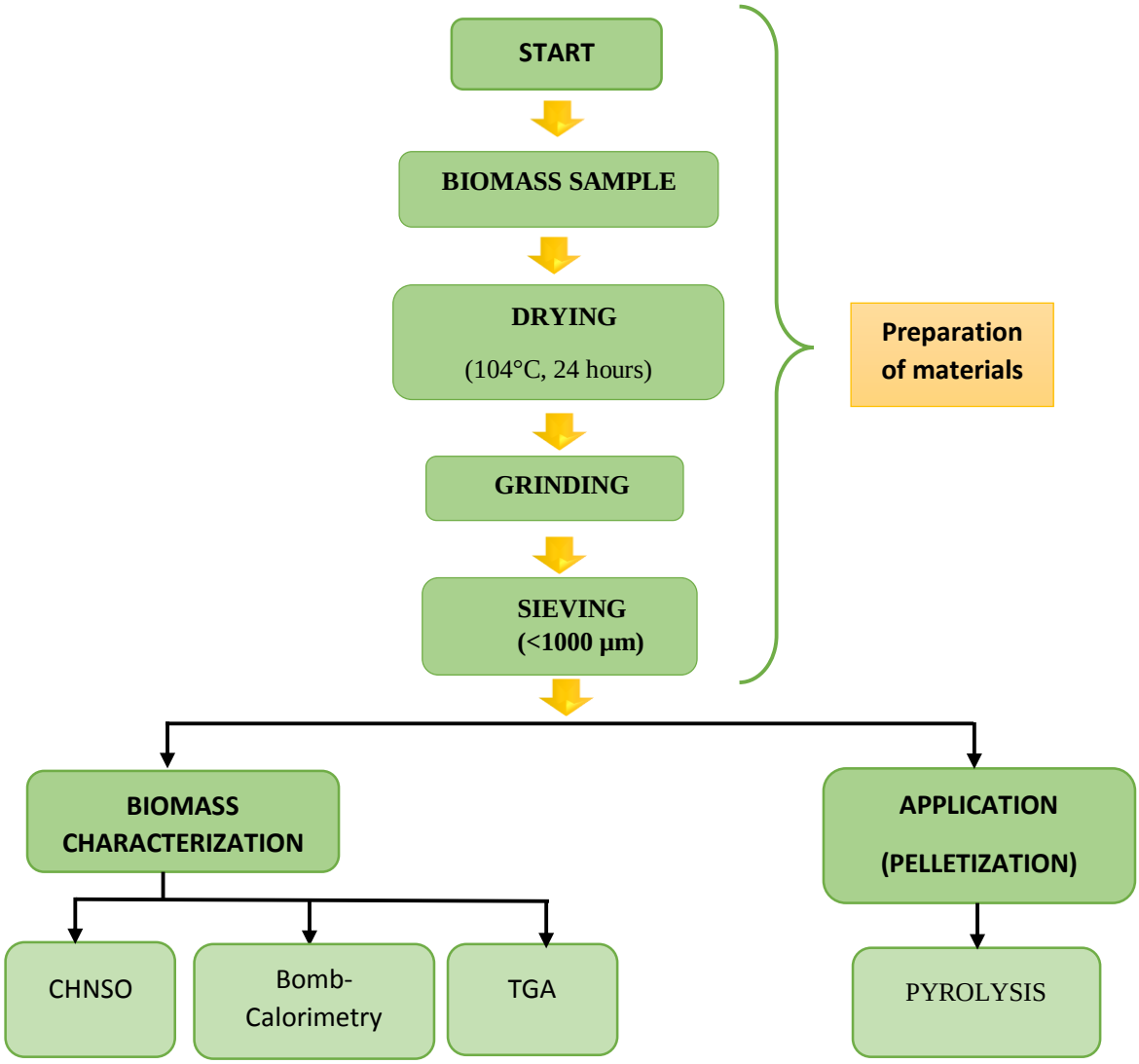

FIGURE 3.1 Project Flow Chart

3.2 Gantt Chart and Key milestones

Table 3.1: Gantt chart and key milestone FYP I

No	Detail Works	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of Project Topic														
2	Preliminary Research Work														
3	Submission of Extended Proposal														
4	Proposal Defense														
5	Project Work Continues														
6	Submission of Interim Draft Report														
7	Submission of Interim Report														

Process Suggested Milestones

All the items in the gantt chart has been successfully completed by the author within the timeline given. For FYP II, the expected detail works that need to be completed in the time frame as shown in Table 3.2 below:

Table 3.2: Gant Chart and Milestone of FYP II

No	Detail Work	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1.	Project Work Continues															
	• Sample preparation (drying,grinding,sieving)															
	• Analysis of materials (TGA, CHNS) and palletization process															
2.	Submission of Progress Report							●								
3.	Project Work Continues															
4.	Pre-SEDEX										●					
5.	Submission of Draft Final Report											●				
6.	Submission of Dissertation (soft bound)												●			
7.	Submission of Technical Paper													●		
8.	Viva														●	
9.	Submission of Project Dissertation															●

3.3 Experiment Methodology

3.3.1 Materials and Apparatus

TABLE 3.3 Material and apparatus used in the experiment

• Woody biomass (saw dust) • Agricultural biomass (PKS) • Drying oven • Grinder • Sieve shaker • Thermogravimetric Analyzer	• Bomb Calorimetric • Pellet Mill • Hydro-cracking Reactor • Mass Spectrometer • CHNSO Instrument

The process will have three main stages which are preparation of materials, biomass characterization and application of the pellet. Each step must be carried out with care if the final product is to be acceptable quality.

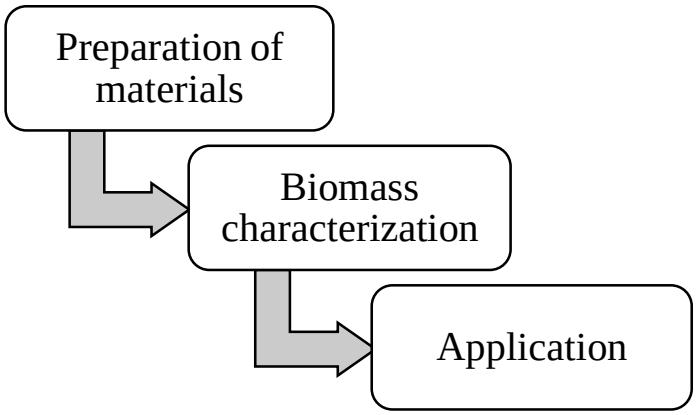

FIGURE 3.2 Main Process of Project

3.3.2 Preparation of Materials

For the preparation of materials, there are three important stages involved as shown in Figure 3.3 below:

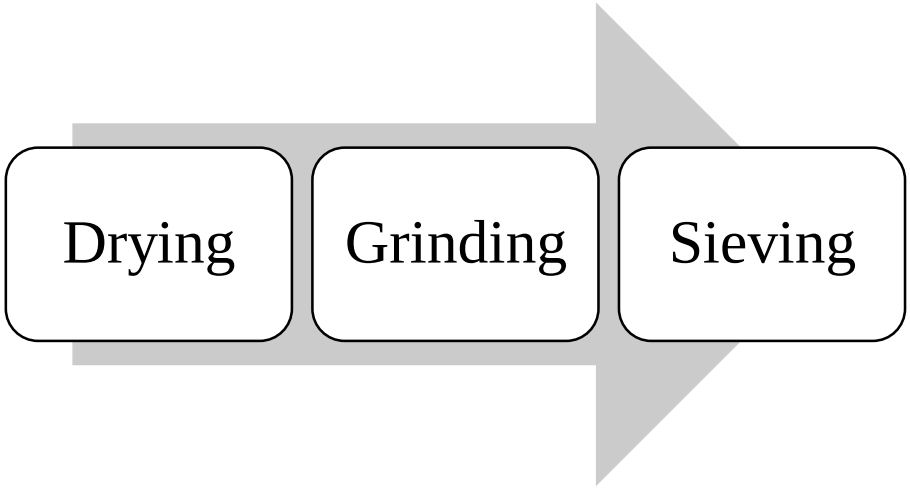

FIGURE 3.3 Preparation of Materials

3.3.2.1 Drying

Two types of biomasses were selected and utilized in this study were saw dust and palm kernel shell (PKS). The sawdust was obtained from the nearby sawmill located at Ipoh, Perak, Malaysia while PKS was purchased from a palm oil mill located in Perak, Malaysia. PKS received were in the form of irregular half-bowl shape. First of all, the biomass samples need to be dried before proceeding with other processes. Both materials were dried in a drying oven from Binder, as shown in Figure 3.4, at 104°C for 24 hours.

From this process, the moisture content of both biomasses can be obtained by simply calculate the percentage of water being removed after drying process. For the overall quality of the final pellets, it is vital to maintain an appropriate moisture level in the feedstock. The required moisture level of the feedstock for wood species was found to be at between 5 to 10 weight percent (Stelte et al, 2012). While for agricultural biomass, several studies show that optimum moisture content for agricultural biomass was found to be at 10 to 15 weight percent. Different levels of moisture content will have different impact on quality in terms of durability and compression stability (Serrano et al, 2011).

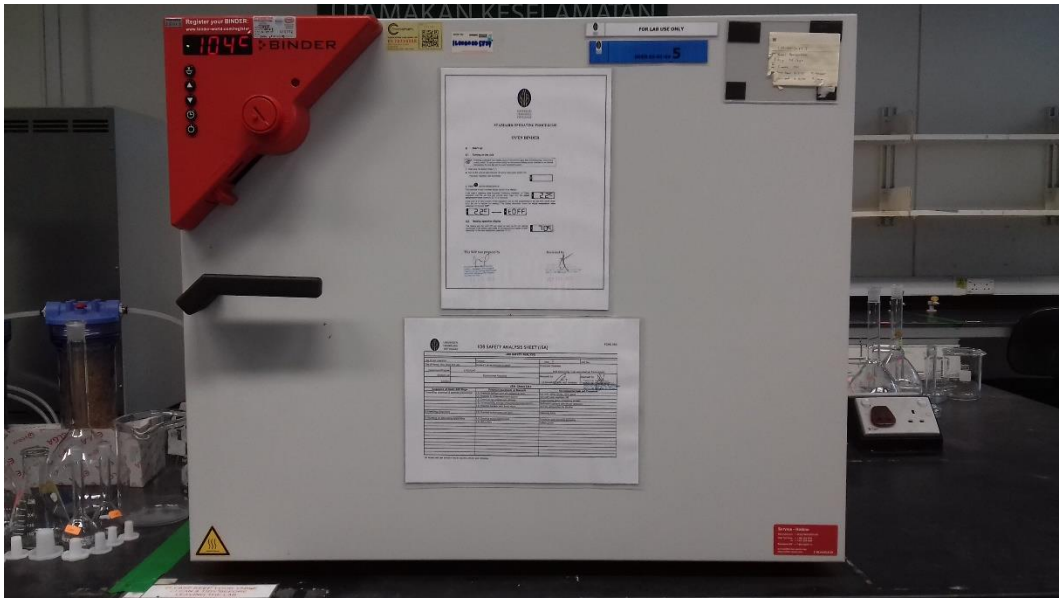

FIGURE 3.4 Drying Oven

3.3.2.2 Grinding

The process then continues with grinding, in order to reduce the size of PKS before pellet compression take place. Saw dust materials did not have to be grinded since the material was already in small size. The densification process will determine the optimum particle size and for pellet production particles are usually below 5 mm in diameter. Particles, which are too small or too large, can severally affect pellet quality and increase energy consumption. The compaction properties of biomass will be influenced by the particle size. The friction in the press channel of a pellet mill decreases with increasing particle size of particles due to decrease in surface area contact between the particles and the channel wall (Stelte, 2011). Firstly, the grinding process took place in a granulator by Shini and grinder by Fritsch as shown in Figure 3.5. Then, sieving process will take place.

FIGURE 3.5 Granulator and Grinder

3.3.2.3 Sieving

Since one of the main focus of this study is to compare the effect of different sizes on the concentration of product gaseous produced during gasification, the samples then need to be sieved. A sieve analysis is a practice or procedure used to assess the particle size distribution of a granular material. A sieve shaker modelled BA 300N, as shown in Figure 3.6, involves a nested column of sieves with wire mesh cloth was used for sieving process. The top sieve which has the largest screen openings was poured with sample. The column was arranged by each lower sieve in the column has smaller openings than the one above. Receiver is a round pan at the base.

FIGURE 3.6 Sieve Shaker

There were three columns with different sizes of opening used for this study. The sizes were 1.0mm,0.71mm, and 0.5mm. The column was placed in a mechanical shaker to shake the column for 20 to 30 minutes depend on the amount of sample placed. After the process complete, the samples will be kept in different air-tight

container based on their respective sizes The sample preparation completed at this stage and ready for the analysis.

3.3.3 Biomass Characterization

Figure 3.7 below shows the biomass characterization involves in this study:

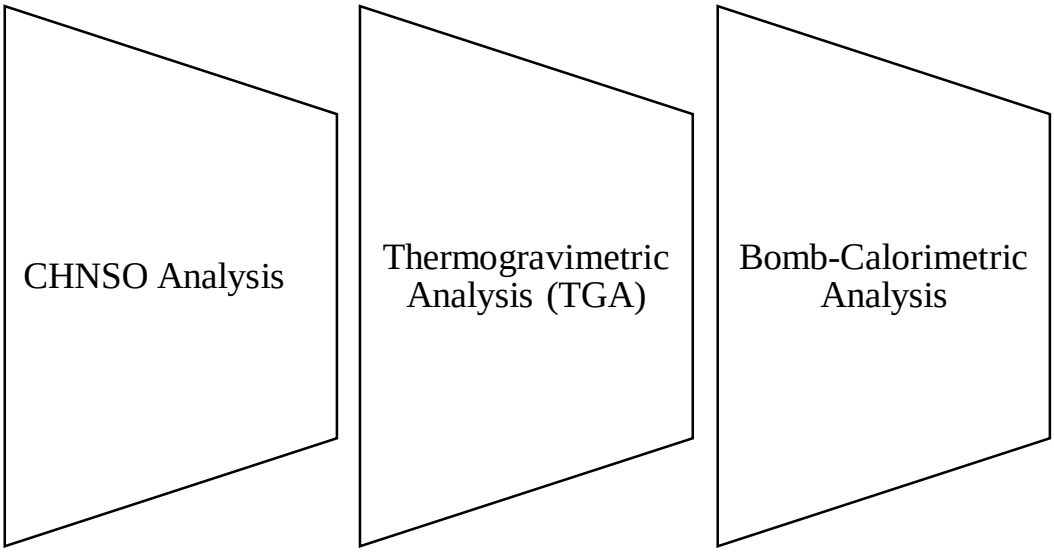

FIGURE 3.7: Biomass Characterization

3.3.3.1 CHNSO Analysis

After the samples have been classified according to their size, this stage will involve chemical analysis of the biomass produced by using CHNSO Instrument and Thermogravimetric Analyzer (TGA). CHNSO instrument is used to conduct ultimate analysis of samples to determine their elemental composition. The results are in percentage composition of Carbon, Hydrogen, Nitrogen and Sulphur. The oxygen composition is determined by subtracting the sum of Carbon, Hydrogen, Nitrogen, and Sulphur compositions from 100.

3.3.3.2 Thermogravimetric Analyzer (TGA)

The Thermogravimetric Analyzer (TGA), model STA 6000 by PerkinElmer as shown in the Figure 3.8 below, is an essential laboratory tool used for material characterization. Various application such as environmental, food, pharmaceutical, and petrochemical use TGA to characterize the materials.

FIGURE 3.8 TGA

It is a technique in which the mass of a substance is monitored as a function of temperature or time as the sample specimen is subjected to a controlled temperature program in a controlled atmosphere. For this study, both woody and agricultural biomass will undergo TGA analysis to determine the weight degradation of the sample with respect to temperature and time in both nitrogen (N_2) and air.

3.3.3.3 Bomb-calorimetric Analysis

In bomb calorimeter, the sample is burnt in pure oxygen within a sealed bomb. The bomb is surrounded by a water bath and the heat of combustion causes a small temperature rise. The bomb calorimetry used as shown in the Figure 3.9 below.

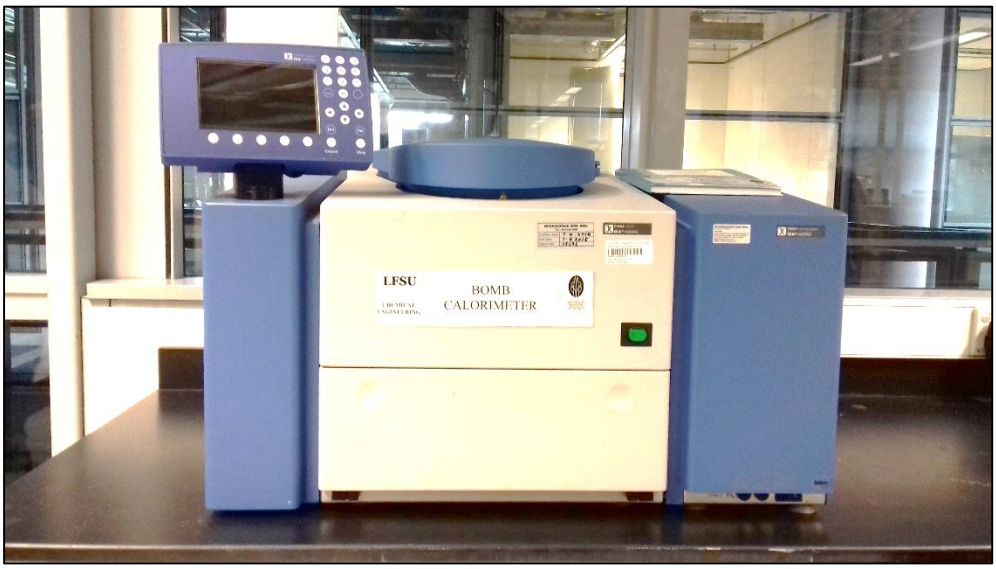

FIGURE 3.9 Bomb Calorimeter

The procedure of bomb-calorimetric analysis started with the main power supply to the bomb calorimeter unit is switched on. Then, the first sample (saw dust) is weighted accurately to 4 decimal point before it is placed into the bomb calorimeter crucible and started measuring its calorific value. The calorific measurement for the first sample is repeated for two times and the average calorific value of it is determined. Experiment is continued with another sample (PKS) and last but not least the main power supply is switched off after the experiment finish.

This analysis is used to calculate an important fuel property which are the higher heating value (also known gross calorific value, GCV) and the lower heating value (also known as net calorific value, NCV) of the sample. GCV of a fuel is defined as the amount of heat released by a specified quantity (initially at 25°C) once it is combusted and the products have returned to a temperature of 25°C, which takes into account the latent heat of vaporization of water in the combustion products while NCV is defined as the amount of heat released by combusting a specified quantity (initially at 25°C) and returning the temperature of the combustion products to 150°C, which

assumes the latent heat of vaporization of water in the reaction products is not recovered (Hydrogen Analysis Resource Centre, 2015).

3.3.4 Application

For the application, the samples will first undergo pelletization process. In some cases, binders or lubricants may be added to produce higher quality pellets by increasing the pellet density and durability. A stabilizing agent needs to be added to agricultural residues since it does not contain much resins or lignin. However, for this study no binders or lubricant is used. Wood contains natural resins which act as a binder. Then, the pellets will undergo gasification process in order to analyzed the product gas produced from the process.

3.3.4.1 Pellet Making Process

After all the material is prepared, the important stage of this study is to produce the pellet by using the pellet mill model ZLSP200B as shown in Figure 3.10. The process of producing fuel pellets involves placing ground biomass under high pressure and forcing it through a round opening called a “die.” The pellet is created in the step known as “extrusion” in which the biomass fuses together to form a solid mass while exposed to appropriate conditions.

FIGURE 3.10 Pellet Mill

A roller is used as shown in the Figure 3.11 below in order to compress the biomass against a heated metal plate called a “die”. The die comprises of several small holes drilled through it that allow the biomass to be squeezed through under high temperature and pressure conditions. The biomass particle will fuse into a solid mass and turning into a pellet if the conditions are right.

FIGURE 3.11 A "die"

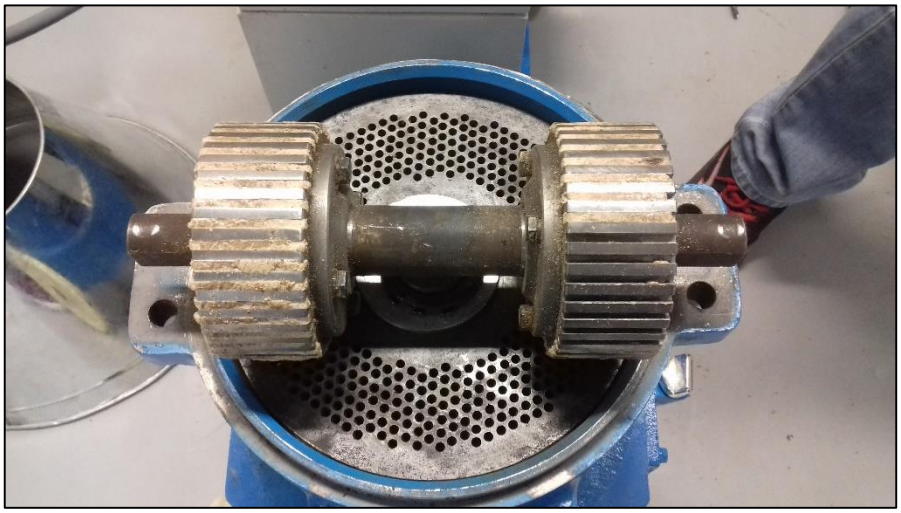

FIGURE 3.12 A roller and a "die"

Before getting started, there are few requirements that need to be considered. Table 3.4 summarize all the requirement as follows:

TABLE 3.4 Requirements for Pellet Mill

Material requirements	
Moisture content	Depends on different kinds of raw material. Moisture content of sawdust is specified 10-18% and the material should be mixed evenly

Requested size	The maximum size of the materials cannot exceed the diameter of the pelletizing hole.
Composition	The mill can process both single kind material and mixture with different materials. Pieces of stone or other hard impurities cannot be mixed otherwise they will damage the die and roller.
Binder	This mill is designed to pelletize without any additive binder. However, binder is recommended which can increase capacity and extend service life of die, roller, and other wearing parts.
Inspection before operation	
Check whether each fastening piece is tightened	It will avoid the bolt coming off and damaging the roller during running.
Check whether safety protection measure is completed	Electric motor, electric cabinet and wires need to be checked before operation to prevent the possibility of electricity leakage.
Adjust the clearance between die and rollers	Clearance between die and roller has great influence on pellet quality. The best range of clearance remains 0.1mm-0.3mm.

A bucket is placed under the discharge outlet so that the material can be collected and put back into the mill several times to preheat the mill. Suitable amount of oily mixture was put into the mill. To ensure the die had been heated enough to produce pellet continuously, there will be a vapor formed above the hopper indicating the temperature in pelletizing chamber is rising. For pelletizing process, the material was feed and the mill was run after preheating. Low moisture content material will produce soft or powdery pellet. Material with high moisture content will produce rough pellet. Adjusting bolt on both side were loosen or tighten if it cannot produce pellets. Different moisture content for both biomasses was tested to get the best pellet quality.

CHAPTER 4
RESULTS AND DISCUSSIONS

4.1 Moisture Content

The drying process takes few runs since the samples need to be dried for 24 hours for each run. From the process, the moisture content can be determined. The formula used to calculate the percentage of moisture content is:

$$\%\ Moisture\ Content = \frac{Initial\ weight - Oven\ dry\ weight}{Oven\ dry\ weight} \times 100\% \quad (1)$$

The test is performed by preparing the sample to be tested, weighing it, drying it to a constant weight, reweighing, and doing the calculation. The result is as in Table 4.1 below:

TABLE 4.1 Percentage of Moisture Content

Type of Biomass	Run	Initial Weight, g.	Oven-dry Weight, g.	% Moisture content
Sawdust	1	251.9705	179.5846	40.3074
	2	398.5425	277.3505	43.6963
	3	384.6717	276.5157	39.1138
	4	351.5843	259.3877	35.5439
	5	419.7277	301.2376	39.3344
PKS	1	378.4053	338.3413	11.8412
	2	514.3367	460.4004	11.7150
	3	477.0532	426.0043	11.9831

	4	714.7588	639.659	11.7405
	5	687.09	616.5713	11.4372

The table is then translated into the graph as in Figure 4.1:

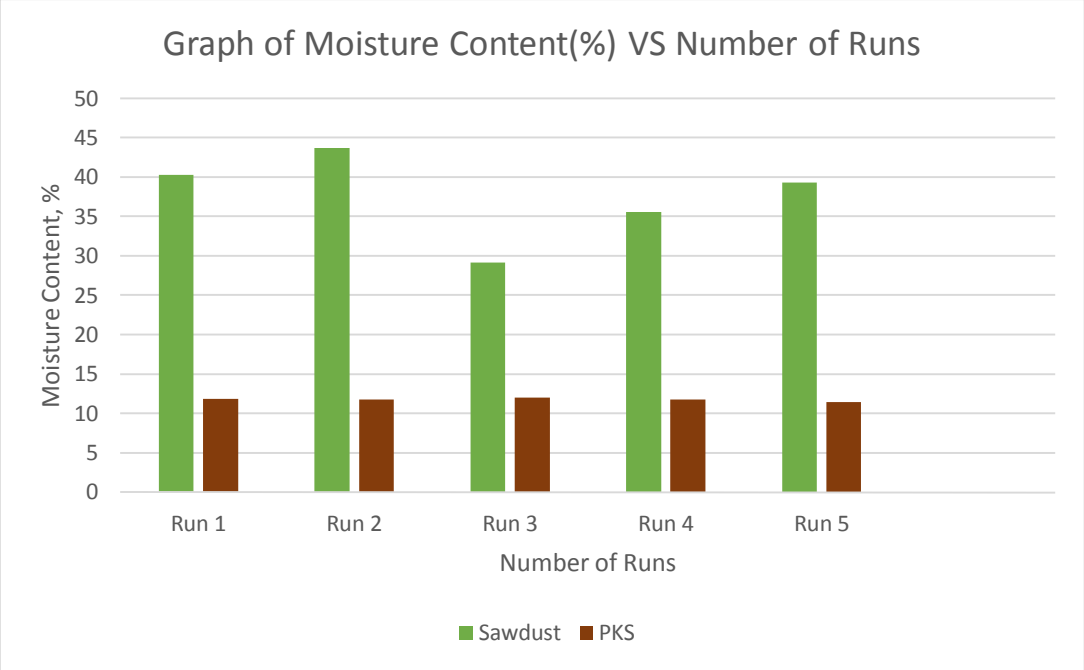

FIGURE 4.1 Graph of Moisture Content VS Number of Runs

From the graph, it can be concluded that moisture content of sawdust is higher compared to PKS. The range of moisture content for sawdust is 35% to 43% whereas for PKS, the moisture content is in the range of 8% to 12% only. It is proven from few studies that state that sawdust will have higher moisture content compared to PKS. From the drying process, the extrinsic moisture content which is affected by weather and is influenced by the surround and humidity has been removed. It is important to identify the moisture content of the biomass to avoid an additional energy penalty related to removal of excess moisture which can also affect the energy produced.

The moisture content before pelletization takes place also has been calculated and the result is as follows:

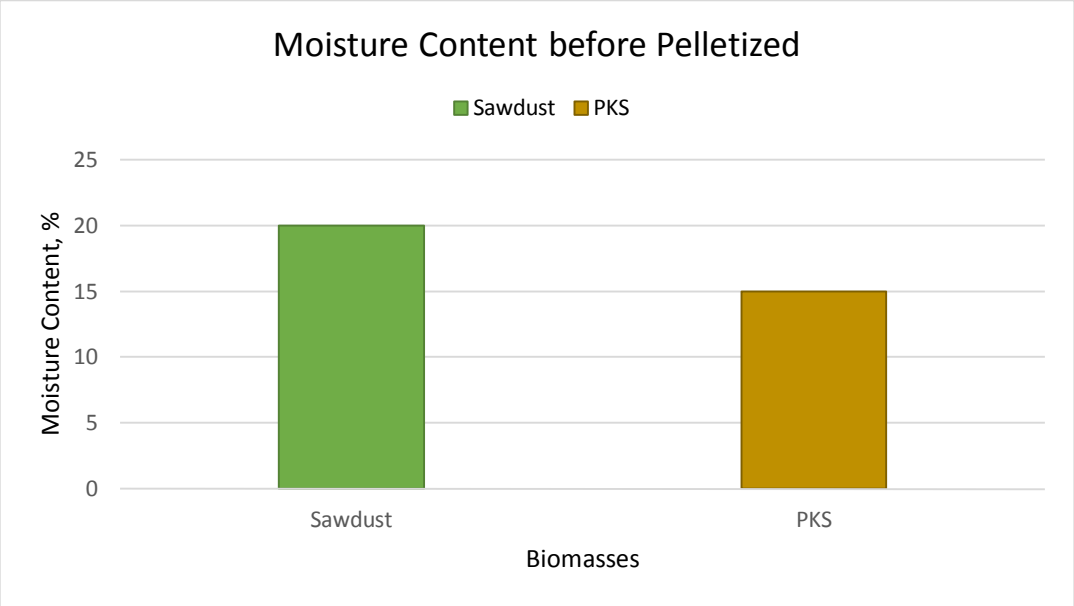

FIGURE 4.2 Moisture Content of Material Before Pelletized

While the moisture content after the materials were pelletized is as in the Fig. 4.3 below:

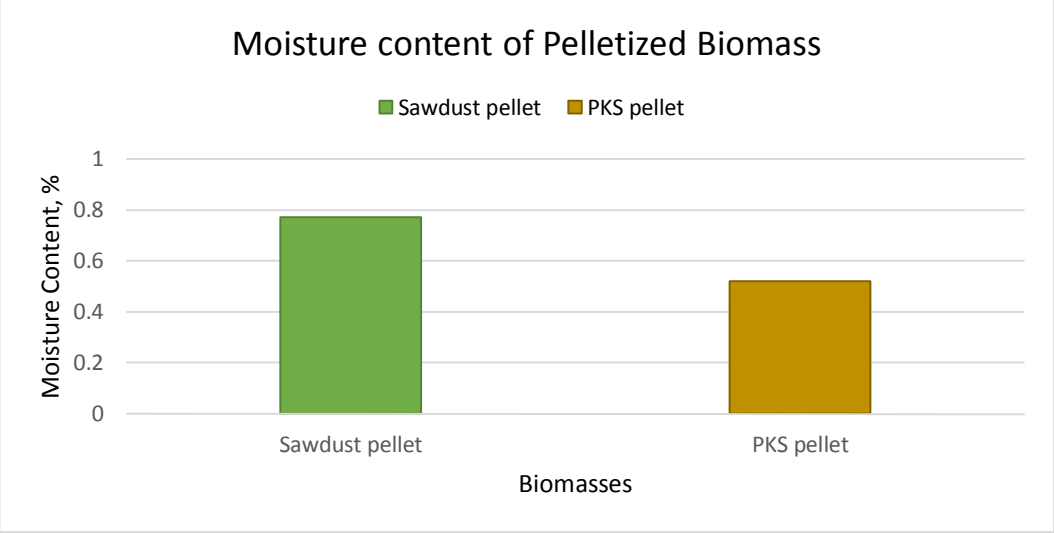

FIGURE 4.3 Moisture Content of Material After Pelletized

Both figures indicate that moisture content of sawdust is higher compared to PKS. This is because PKS basically did not have pocket to store water. Therefore, its

moisture content is lesser than sawdust. While for pelletized biomass, the moisture content is not the same as before pelletized. It decreases as during pelletization process, some moisture has been extracted out due to compression and friction which produce heat at the same time. Thus, it is justified why does moisture content of both materials decreases.

4.2 Proximate Analysis

Based on proximate analysis, the ash content, volatile matter, moisture and fixed carbon are determined. moisture content is calculated in the section above. To evaluate the ash content in biomass, 1.0g of PKS and sawdust being burn until no more smoke or flame appears by using ashing burner and clay triangle with stand. Then they are heated up in muffle furnace at 575℃± 25℃ for 12 hours to assure complete burning of carbon in the samples. The sample is then cooled and weighed. The percentage of volatiles matter is extracted from TG results. Fixed carbon is determined by subtracting sum of volatiles matter, ash content and moisture content. The results of proximate analysis are given in Table 4.2.

TABLE 4.2 Proximate Analysis

Parameter	**PKS (composition, wt%)**	**Sawdust (composition, wt%)**
Ash content	4.30	0.07
Volatiles matter	73.12	54.98
Moisture content	11.00	35.00
Fixed carbon (by difference)	11.58	9.95

The results indicate that PKS contain high ash content compared to sawdust as it might contain greater alkali matter or inorganic compound. The same trend goes to volatiles matter and fixed carbon. The concentration of cellulose, hemicellulose and

lignin in PKS is much higher than sawdust and thus this will affect in the energy value and weight degradation with respect to temperature in TG analysis.

4.3 Ultimate Analysis / CHNSO Analysis

Another way to present the components in the organic part of fuels is from the elemental or ultimate analysis, which commonly referred to as the CHNS analysis. The main elements present in the organic part of biomass is presents directly from ultimate analysis. The results are represented in two different figures to make the comparison easier as the percentage different ranging from 49% to 0.2%. Figure 4.4 below shows the result of the analysis for the elements of carbon and hydrogen.

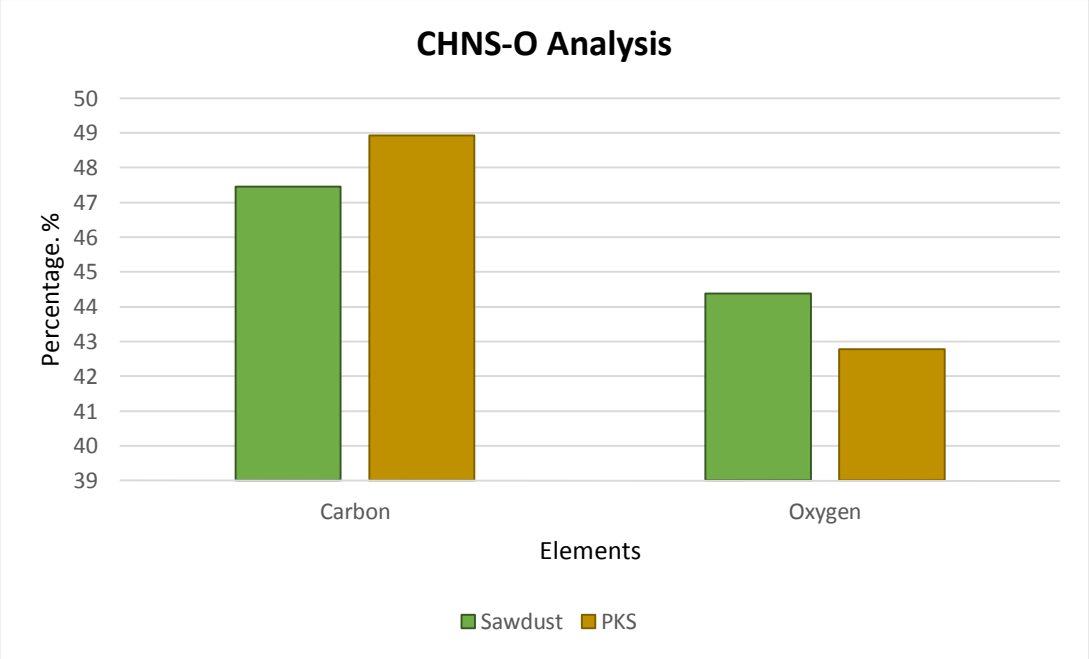

FIGURE 4.4 Result of CHNS-O Analysis (Carbon & Oxygen Elements)

From the result, the most important constituent for both biomasses fuels is obviously carbon. This is because they capture atmospheric CO_2 for photosynthesis process and that became part of the plant matter. Meanwhile, carbon is mainly transformed back into CO_2 during combustion, which is again released in the

atmosphere and thus create the carbon cycle. Carbon also represents the major contribution in the overall heating value. The content of lignin, hemicellulose and cellulose will affect the carbon content of the fuel. From this study, PKS has higher lignin content leads to a higher carbon content as compared to woody biomass. This carbon content also will contribute to higher heating value of PKS.

The second major element present in both biomass fuels is oxygen. For this case, oxygen content in sawdust is much higher compared to PKS. This is resulting from the chemical composition of the biomass constituents and the nature of photosynthesis process. Fuel oxygen which is chemically bound in the molecules of CO_2 and H_2O is found in the combustion products. The amount of air needed for combustion is reduced because of this. It should be noted that weight concentration of oxygen is calculated by difference. In order to get the weight concentration of oxygen, the concentrations of all other elements (C, H, N, S) is subtracted from 100. While for the other three elements such as Hydrogen, Nitrogen and Sulphur, their constitution is lower compared to Carbon and Oxygen. Thus, the result is represented separately in Figure 4.5 below:

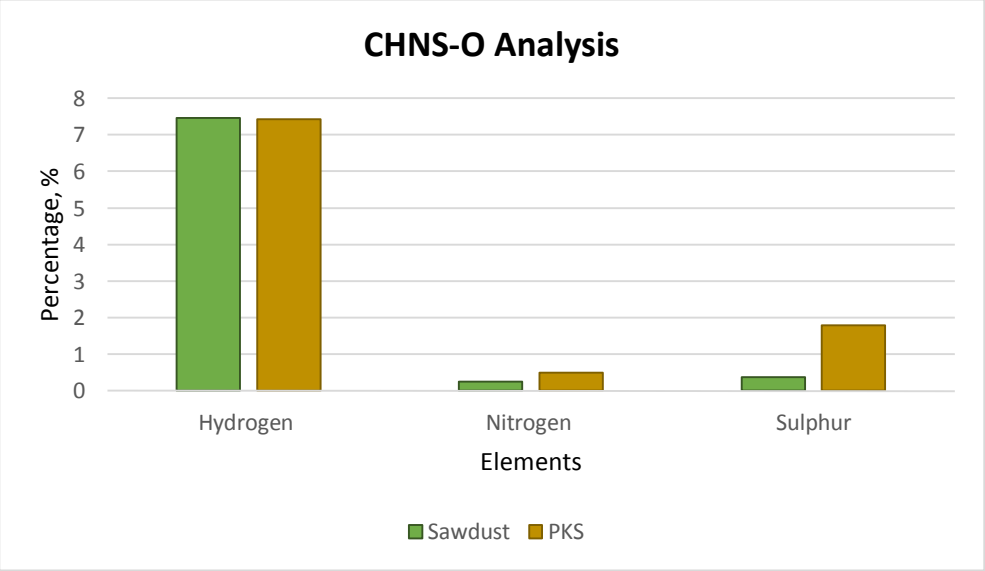

FIGURE 4.5 Result of CHNS-O Analysis (H_2, N_2 & Sulphur Elements)

Another major constituent of biomass is hydrogen, as can be expected from the chemical structure of the carbohydrate and phenolic polymers. Hydrogen is converted to H_2O during combustion, significantly contributing to the overall heating value. The weight content of hydrogen in PKS is slightly lower compared to sawdust. For nitrogen, the most important nutrient for plants which is absorbed through the soil or the applied N-fertilizers by the plant during its growth. PKS species have a higher N content compared to woody biomass types due to their high growth rate and the application of fertilizers. N_2 contribution to the overall heating value is zero as during combustion and for all practical purposes, nitrogen does not oxidize in any significant quantities and is released in the gas phase as N_2.

Along with nitrogen, phosphorus and potassium, Sulphur is an important nutrient for plant growth which is incorporated in several organic structures like amino-acids, proteins and enzymes. Its concentration in PKS is also slightly higher compared to sawdust as the high growth rate of agricultural biomass. Compared to most coals and certain types of liquid fossil fuels, the Sulphur content of these fuels is still lower. Sulphur has a minor contribution to the overall heating value as during combustion, it is typically oxidized. Percentage of Sulphur will also contribute to the production of ash. Higher Sulphur content will increase the percentage of ash formed. It is proven as from the proximate analysis, the ash content of PKS is much higher compared to sawdust.

4.4 Thermogravimetric Analysis (TGA)

Thermal Analysis (TA) is a group of techniques that study the properties of materials as they change with temperature which includes several different methods. These are distinguished from one another by the property which it measured. For this study, the author used Thermogravimetric Analysis (TGA) which measured mass degradation with respect to temperature. The analysis was conducted from room temperature to 800°C. The result is as shown in the Figure 4.6 and Figure 4.7 below.

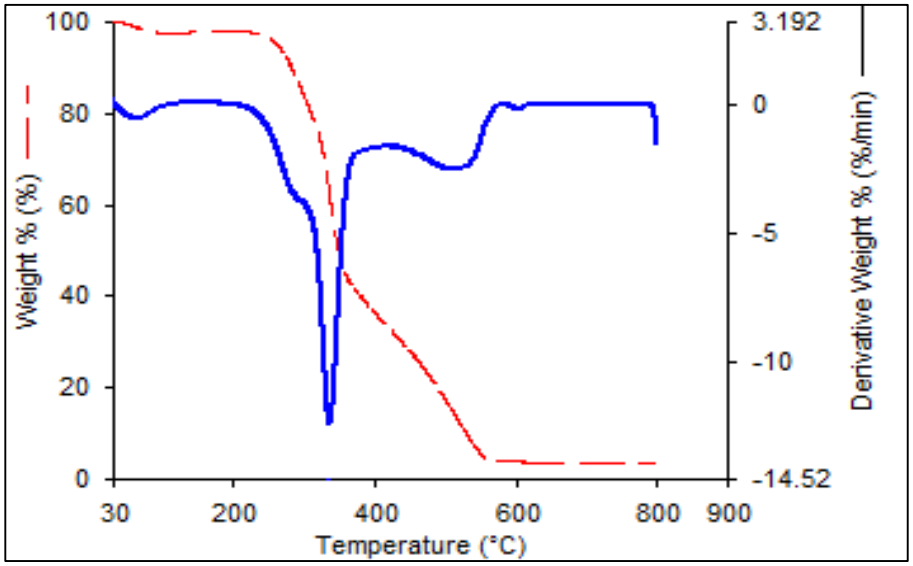

FIGURE 4.6: TGA analysis of sawdust

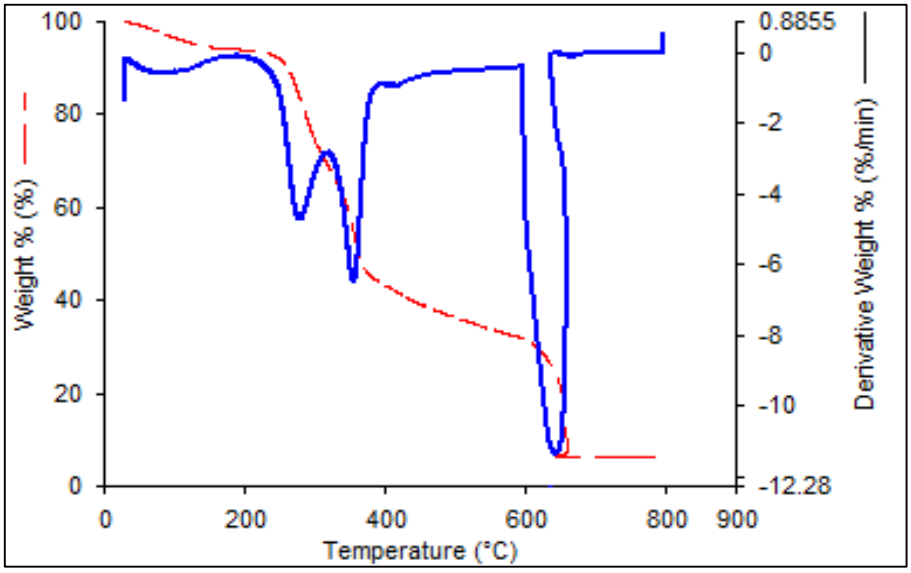

FIGURE 4.7: TGA analysis of PKS

TGA graphs produced on the computer as a result of analysis provides the detailed information about the mass loss of biomass against different temperature. The shape of TG curve as seen in Fig 4.4 and Fig.4.5 show the single stages of decomposition. The behavior of DTG curve line against the temperature of both biomass is shown in Figure 4.5 below. In spite of materials tested, a TG curve can distinguish three different particular regions. Generally, TG curve will display the weight change of a sample with respect to time or temperature. The thermal behavior

of solid fuels in pyrolytic conditions is the information that we can get from this curves. On the other hand, DTG emphasizes the zone of reaction where various reaction steps are taking place over the entire temperature range.

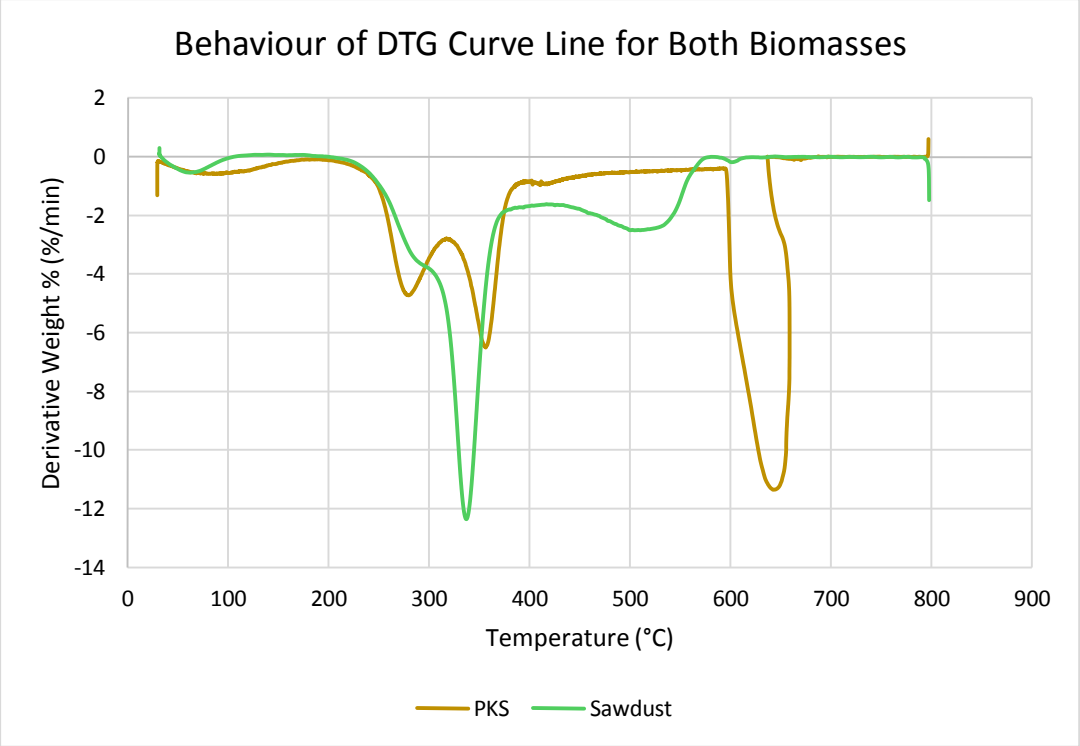

FIGURE 4.8 Behavior of DTG curve line for both biomasses

The difference between final temperature point and initial temperature point indicates reaction interval. Within this range biomass lost most of its weight. When the biomass undergoes pyrolysis, the main components of biomass such as carbohydrates (hemicellulose, cellulose), lignin and extractives, are separately degraded as they have separate thermo-chemical behavior. At the temperature up to 200°C, the shape of TG curve shows desorption or drying which means both biomasses will lose its moisture content and light volatiles. In the first stage temperature up to 100°C all biomass lost their mass in the range between 5-10% as evaporation of moisture content. Sawdust shows greater loss mass compared to PKS due to its higher moisture content.

In the second stage of temperature ranges between 250°C-450°C all biomass lost their most of the mass which indicates value of volatile matter content burned.

TGA graph suddenly drops down fast in this range of temperature. All the volatile matter content is burned approximately in the temperature between 250°C to 400°C. Both TGA graphs for sawdust and PKS shows significant drop at temperature 250°C to 350°C. Steeper slope can be seen for sawdust which indicate greater amount of hemicellulose and cellulose present in the biomass compared to PKS.

Similarly, two main reaction regions during the pyrolysis of sawdust and PKS were observed from DTG profiles. The first region refers to devolatization of biomass that took place at temperature around 200 °C due to combustion of light volatiles present in the biomass. As observed in Figure 4.7, two ‘‘shoulder peaks” occur in the first region of the DTG curve at around 280^{o}C and 350 °C, respectively, for PKS. This peak could be attributed due to the decomposition of hemicellulose and cellulose, respectively. On the contrary, only one peak was observed in sawdust. This could possibly be due to their higher hemicellulose content than cellulose, which caused the reaction mechanism to be just one peak. Another reason could be due to delay in the thermal decomposition of the hemicellulose.

The analysis continues for the third stage which in the temperature range of 400°C to 650°C. for PKS, steeper slope showing high amount of lignin being liberated throughout this range of temperature. From this result, it can be concluded that lignin content in PKS is higher than sawdust which will affect the ash content and energy value of the biomass. Complete combustion occurred for PKS at the temperature of 650°C as can be seen from the graph. While for sawdust, it has already completely burned before reaching 600°C.

4.5 Bomb-Calorimetric Analysis

To determine the energy value of biomasses, bomb-calorimetric analysis is conducted and the result is in the Table 4.3 below.

TABLE 4.3 Energy Value

Sample	Weight, g	Energy J/g
Dried PKS	0.6195	20414
Dried PKS	0.6644	20263
PKS	0.5311	16194
PKS	0.5335	13600
Dried Saw Dust	0.5583	19464
Dried Saw Dust	0.6058	19969
Saw Dust	0.4266	14549
Saw Dust	0.4604	15251

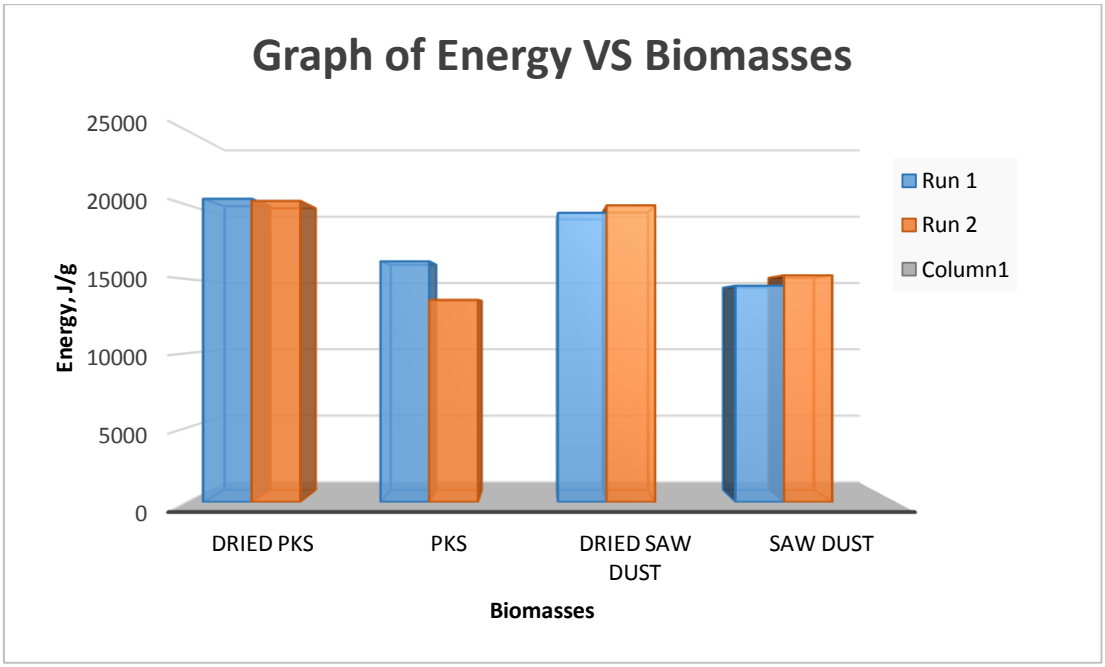

FIGURE 4.9 Graph of energy VS biomasses

From the result above, it shows both dried PKS and dried saw dust produced higher energy compared to PKS and saw dust. High moisture content in sawdust will have a much lower net energy density by mass, owing to the weight of the water, but also by volume owing to the energy required to evaporate the water. The result also

shows that PKS have higher energy value compared sawdust. It is also a significant effect of moisture content. When the material has lower moisture content, it indicates that the components have occupy the space inside the biomass and significantly will increase the energy produced.

4.6 Pelletization

Both biomasses, sawdust and PKS undergoes pelletization by using the pellet mill. Several moisture content was tested to give the best quality of pellet. From the research, the best moisture content for PKS is 20wt% while for sawdust is at 25wt%. There are few factors affecting the production of pellet such as type of die used and the size of particle. The result of pelletization process are as in the Figure 4.10 and Figure 4.11.

FIGURE 4.10 Pelletization of Sawdust

FIGURE 4.11 Pelletization of PKS

The pellets produce will undergo pyrolysis process in order to collect the gas produced. The gas collected will be analyzed by using GC to determine their concentration.

4.7 Product Gas

The product gas is collected at the temperature of 350°C, 450°C and 540°C during pyrolysis process and the concentration is being analyzed by using Gas Chromatography (GC). The results are as in the four consequent figures below.

4.7.1. Species of Biomass

As stated in objectives, the research is to study the concentration of product gas for two different species which are PKS and sawdust. Thus, the first two graphs, which are the concentration of product gas for PKS while the other two graphs are for sawdust. From these graph, the trend of concentration of product gas are almost similar, in which, the three gases, CO_2, CH_4 and H_2 are increasing up to temperature of 450°C then starts to reduce. While for O_2, it behaves the other way around as it decreases first up to temperature almost the same as the other three gases and then starts to increase the concentration as the sample has been oxidized. Different species of biomass will have a different lignocellulose material and can affect their

performance. But for this case, PKS and sawdust act almost similar in the concentration of product gas. Therefore, it can be said that agricultural waste, PKS, can perform as great as woody species, sawdust.

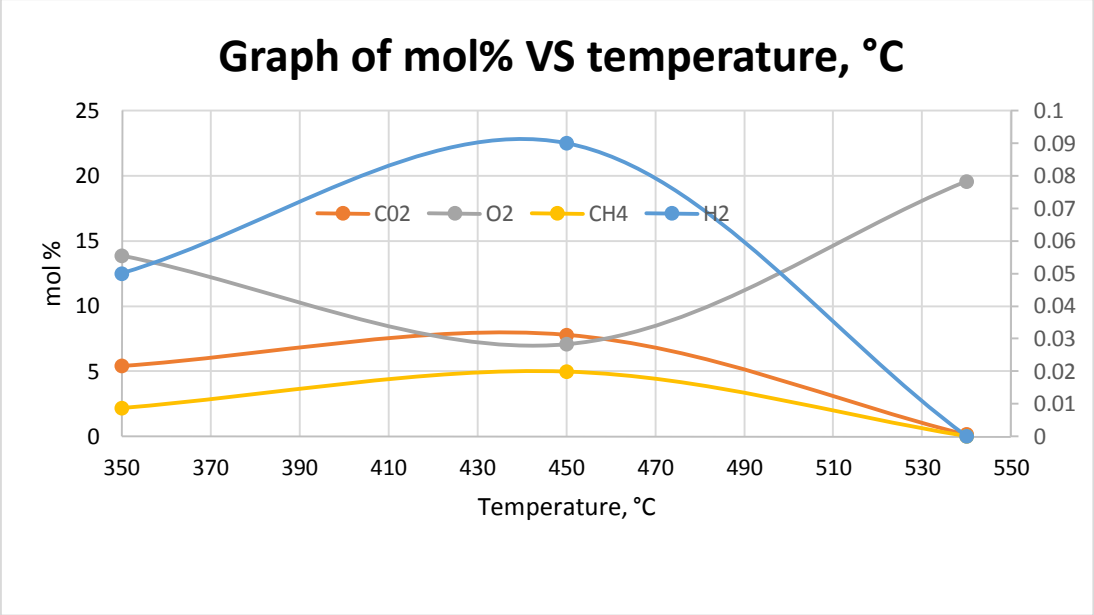

FIGURE 4.12 Concentration of product gas for PKS <0.5mm

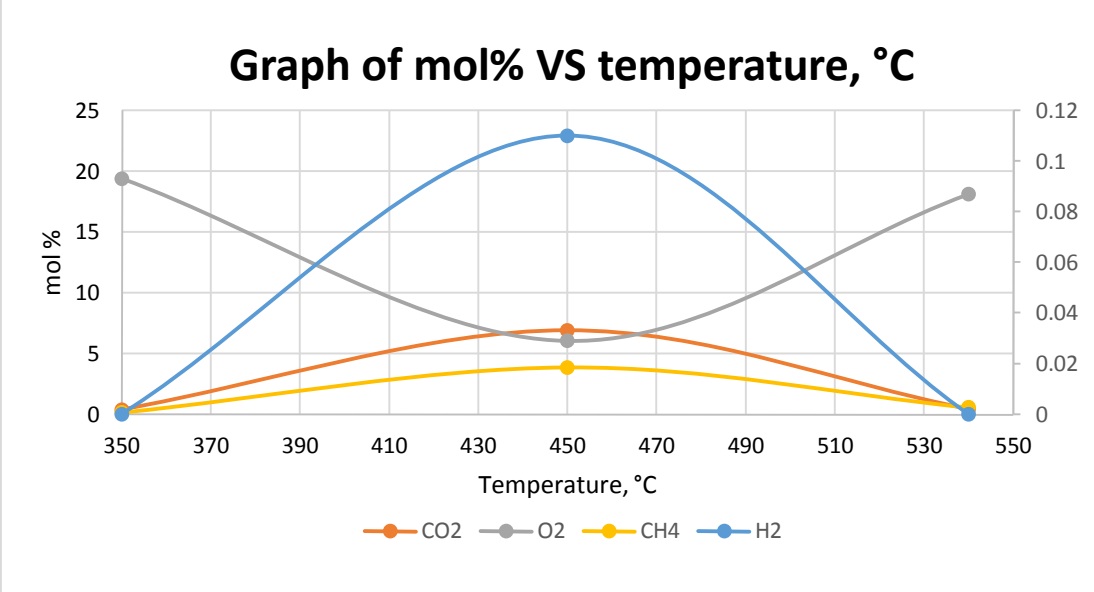

FIGURE 4.13 Concentration of product gas for PKS <1.0 mm

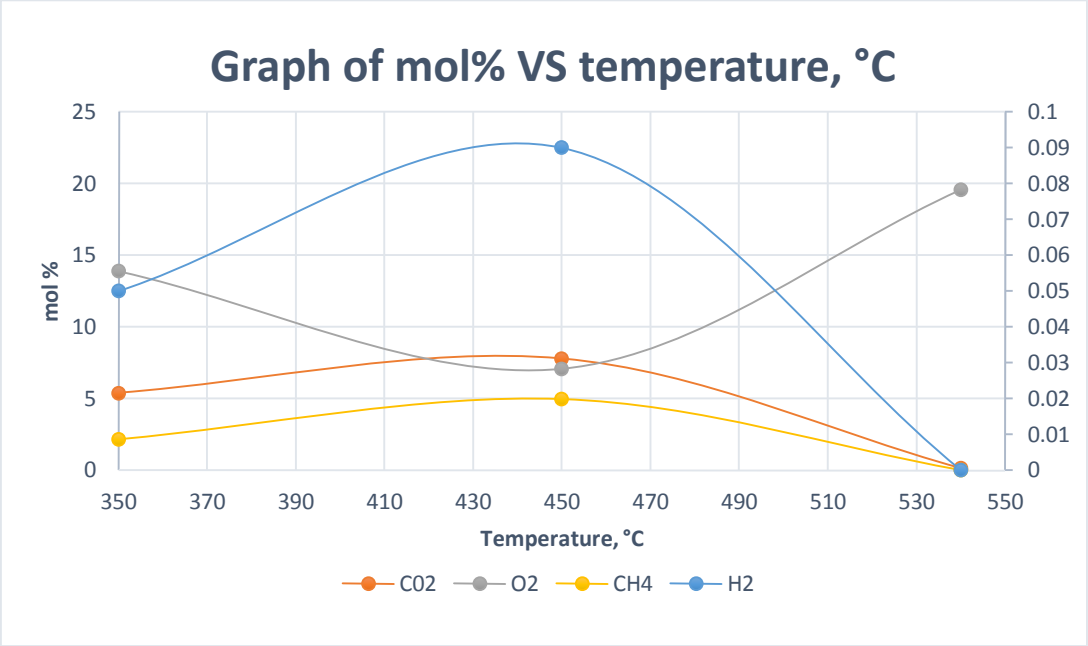

FIGURE 4.14 Concentration of product gas for sawdust <0.5 mm

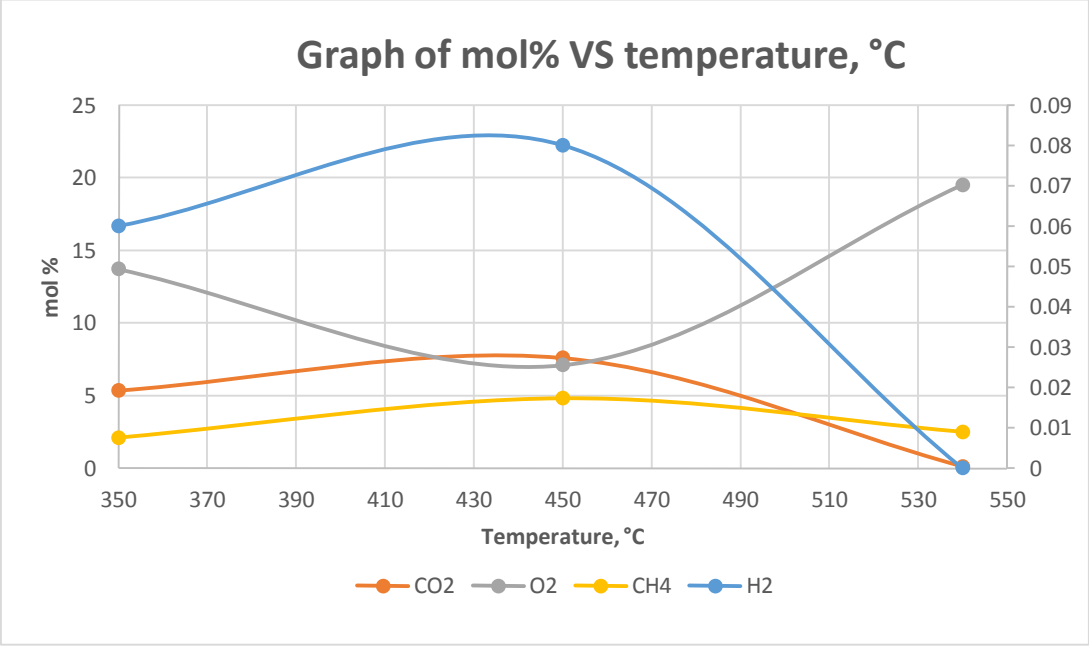

FIGURE 4.15 Concentration of product gas for sawdust <1.0 mm

4.7.2 Temperature

The concentration of product gas varies significantly for the three temperatures. It can be said that temperature plays an important role in pyrolysis process and affect the gas produced. In addition, both composition and yield of product is determined by pyrolysis temperature (Basu.P,2013). From the graph, we can see that the concentration of product gas varies with temperature. This is because the decomposition of lignocellulose material occurs at different temperature. For instance, the decomposition of cellulose is at temperature range of 300°C - 400°C while for hemicellulose, the decomposition started at the temperature of 200°C - 300°C as it is lack of crystallinity and the presence of acetyl group (Yang et al.,2007) that cause it to be least stable component. For lignin, it is thermally stable compared to cellulose and hemicellulose and it has broader decomposition temperature from 280°C - 500°C and the maximum release rate is at 350°C - 450°C. The gas is being collected at the temperature of 350°C, 450° and 540°C to indicate the effect of decomposition of biomass material on the concentration of gas produced.

Therefore, the concentration for CO_2, CH_4 and H_2 is lower at the temperature of 350°C compared to temperature of 450°C for both biomasses. It can be said that the decomposition of cellulose and hemicellulose has took place at this range of temperature. The concentration of the gas keeps on increasing as the decomposition of lignin also is considered to happen at this range of temperature. At 450°C, all the three gases start to decrease in concentration for both biomasses while for PKS with the particles less than 1mm, this is the maximum concentration achieved due to the effect of particle size which will be discussed in the section below. After the species reached the maximum concentration of gas produced, they started to decrease the concentration as all the components has been fully decomposed. As can be seen from the graph, when the temperature is increased up to 540°C and the gas is collected, the concentration shows the lowest concentration and almost to zero mol%. While for O_2, it behaves contradictory compared to the other three gases as can be seen from the graph. The concentration decrease first as O_2 is used for the process and increase again as the

temperature raised indicating the biomass has been oxidized. This result also is comparable with TG Analysis that shows decomposition of cellulose, hemicellulos and lignin with respect to temperature.

4.7.3 Particle Size

Another parameter of interest is the effect of particle size of biomass to the production of gas. As for sawdust, there is not much different on the concentration of the gas produced between these two particle sizes, less than 1.0mm and less than 0.5mm. Both graphs show almost similar trend, increase up to 430°C and then decrease. As for O2, it behaves contradictory as explain in the section above.

While for PKS, there is a slight difference at the temperature of 350°C. At this temperature, the concentration of the product gas for particle size of less than 0.5mm is higher compared to particles size of less than 1.0mm. For particle size less than 1mm, the concentration for CO_2, CH_4 and H_2 is 0 mol% while for the particle size of less than 0.5mm, these gases have a concentration around 3 to 13 mol %. This resulting from the condensable gas facing more resistance to escape easily to surroundings when the particle size is bigger and thus needs more energy to overcome the resistance. The maximum concentration of product gas for less than 1mm size is at 450°C while for particle size less than 0.5mm, the maximum concentration of product gas is around 430°C. Therefore, it can be said that particle size can affect the time and temperature to produce the product gas for PKS. The bigger the particle size, more energy is needed and thus increase the temperature for the product gas to be formed.

CHAPTER 5

CONCLUSION

As a conclusion, all the three objectives of this research is completely done. For the characterization of biomass, both species has been analyzed accordingly by conducting proximate and ultimate analysis, energy value, and also TG analysis. High percentage of lignin content in the biomass will affect the energy value, which can be seen from the bomb-calorimetric analysis showing PKS with higher energy compared to sawdust. The ash content also is affected by the lignin content as PKS showing greater amount of ash. TG analysis also shows a steeper slope for PKS at the temperature where lignin is supposed to decompose indicating greater amount of lignin present in PKS. Then, degradation profile of both biomasses were compared. This result is used to compare with the proximate and ultimate analysis conducted. Each region represents different decomposition of materials with respect to temperature. Two 'shoulder-peaks' of PKS formed could be attributed due to the decomposition of hemicellulose and cellulose, respectively.

Next objective is to compare the concentration of the product gas from two different sources of biomass. The results show almost similar trend for both PKS and sawdust but the parameter such as temperature and particle size will affect the gas produced. The concentration of gas produced (CO_2, CH_4 and H_2) will increase perpendicularly with temperature up to around 430°C and will decrease as all the material has been fully decomposed. However, O_2 gas will act differently as it decreases first then starts to increase as the material has been oxidized. The effect of particle size can be seen for PKS as bigger size of particle needs higher temperature to produce the product gas. More energy is needed for the gas to overcome the obstacles

and escape to surrounding. While for sawdust, the particle size has not so much effect on the production of gas. 10mg of powder tested using TG perform similar in the chemical characteristics and product gas, as compared to 10g of pellets tested in hydro-cracking reactor. Although there is significant effect of the particle size for PKS as discussed in the result, the performance of PKS to be used in energy generation system is similar with the performance of sawdust, biomass from wood. Therefore, further study is needed for utilization of agricultural waste since there are a number of technological options available to make use of a wide variety of biomass types as a renewable energy source. This will increase the percentage of world's energy budget for biomass and consequently reduce our dependency to fossil fuel and thus protecting our environment from global warming.

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